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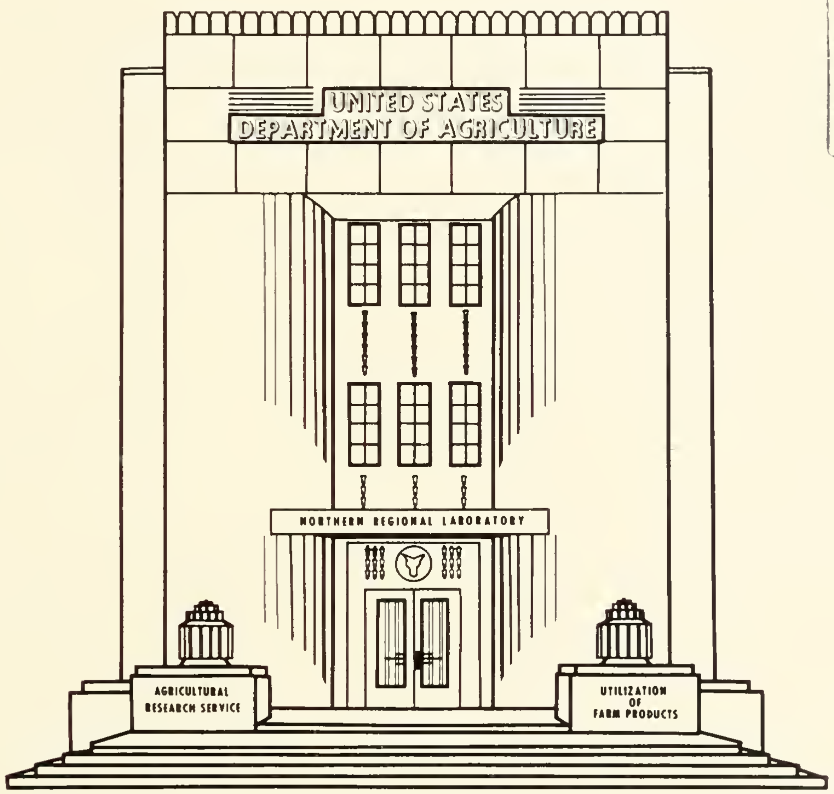
BRIDGING UTILIZATION ENGINEERING DEVELOPMENT TO INDUSTRIAL USAGE



Agricultural Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

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BRIDGING UTILIZATION ENGINEERING DEVELOPMENT TO INDUSTRIAL USAGE[1]

By

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In 1938 the U.S. Congress authorized the establishment of four regional research laboratories, one in each major agricultural-producing area, to conduct basic and applied research designed to expand, improve, and develop new, scientific, chemical, and technical uses for American farm crops and to extend markets for their products and by-products. The four laboratories, now Divisions of the Agricultural Research Service of the U.S. Department of Agriculture, are located at Peoria, Ill.; Philadelphia, Pa.; New Orleans, La.; and Albany, Calif.

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[1] This paper was presented as part of a symposium on "Chemical Engineering in Broadening the Use of Agricultural Crops," sponsored by the American Institute of Chemical Engineers at its 53d annual meeting in Washington, D. C., December 4-7, 1960.

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Figure 1.--The Northern Regional Research Laboratory at Peoria, Ill., is headquarters for the Northern Utilization Research and Development Division.

The Northern Division at Peoria has a full-time staff of approximately 400, of which more than half may be classified as professional research people (figure 1). They conduct chemical, chemical engineering, and other scientific research to develop new uses for the cereal grains--corn, wheat, grain sorghum, and oats; and the oilseeds--soybeans, flaxseed, and erucic acid-containing oilseeds. They also screen new crops to determine if they offer possibilities as replacements for crops currently produced in excess of consumption. The work is approximately 40 percent basic research and 60 percent applied research; however, this ratio can be varied depending upon the urgency of current projects.

In this paper the importance of transferring chemical engineering developments of utilization research to industrial usage is discussed and illustrated.

The position of the Northern Division is unique in that it will never be a manufacturer or a consumer of its developments. Our basic goal in the utilization of agricultural crops is realized when an industrial company uses and commercializes our developments. Information on developments is published, and, if possible, processes and products are patented. All patents become public property and are available for licensing without charge. No company or groups of companies are favored or given exclusive rights.

Most engineering projects originate in the chemical laboratories. Some projects that are primarily engineering problems evolve from work within the Engineering and Development Laboratory itself. Others result from needs reported by various processing industries or agricultural groups. Our pilot-plant studies provide:

1. Development of commercially feasible processes.

2. Information on types of equipment needed.

3. Basic engineering data required for the design and operation of commercial units.

4. Data to predict with reasonable accuracy what a new product will cost.

5. Product for setting specifications and evaluation.

Since the Northern Division's engineering, operating, and other personnel will not be available on a continuing basis at the ultimate industrial user's plant, our pilot-plant work must be very thorough, with no loose ends, and must be fully reported. In addition, there may be detailed discussions with the ultimate user or visits to his plant, but from a long-range standpoint, personal contact is primarily advisory and definitely is not a permanent arrangement. In commercialization of our developments, there can be no place for opinions or on-the-spot decisions often found in industry when they are working on their own developments.

The scale of our pilot-plant operations varies widely from project to project, and at various stages production may range from a few pounds to thousands of gallons. In general, our studies are made on the smallest scale that will provide adequate chemical engineering data although, as in industry, the size chosen must often be a compromise based on the facilities and personnel available.

Primarily our main pilot-plant facilities include one wing of the main building proper, divided into two major work areas: a separate fermentation semiworks building and a separate cereal dry-milling laboratory. Pilot equipment representing practically all the engineering unit processes and most types of processing equipment are available. Typical views of the pilot-plant facilities are shown in the following figures. The main pilot plant is about 175 feet long by 64 feet wide and 45 feet high from the floor to the roof beam (figure 2). The center area is generally open and there are two mezzanines around each side (figure 3) and on one end. Equipment is located on each deck. An enclosed area contains a complete soybean solvent-extraction plant.

Back of this main area is an additional room, 80 feet long by 64 feet wide, in which are located an alcohol pilot plant and other experimental equipment. A general one-directional view of our grain cooking, fermenting, and distilling equipment is shown in figure 4.

Figure 5 is a view of the pilot vegetable oil deodorizer in the oil-extraction plant. Work on soybeans at the Northern Division has contributed greatly to the development of soybeans into one of the most important U. S. crops.

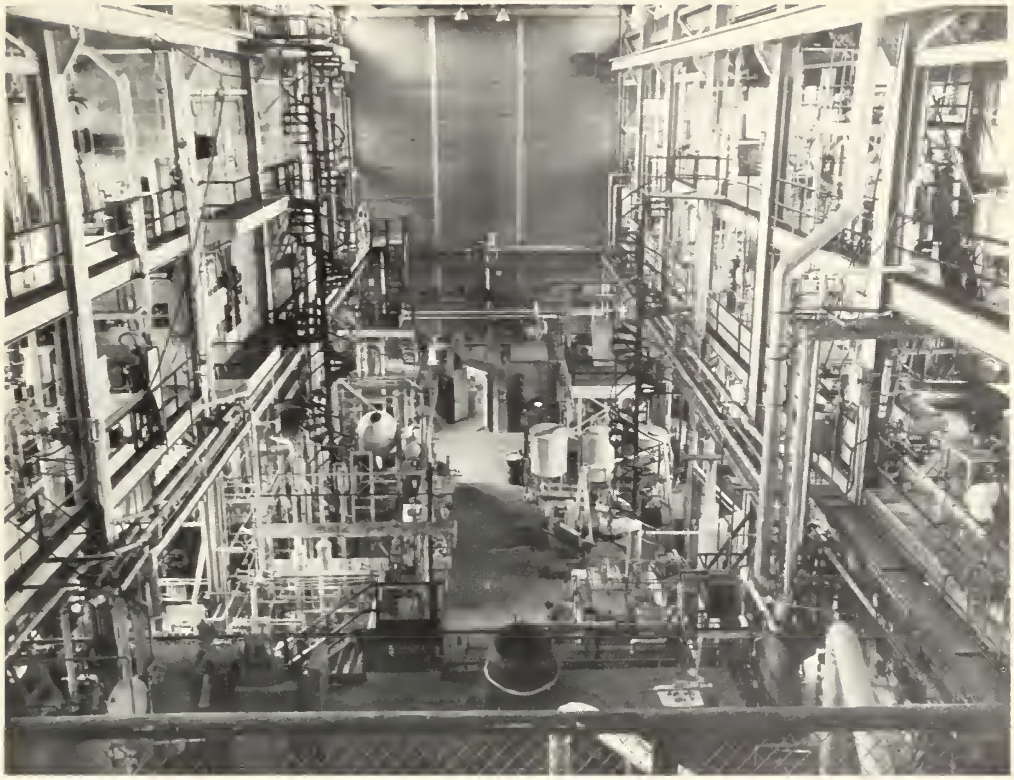


Figure 2.--Main pilot plant.

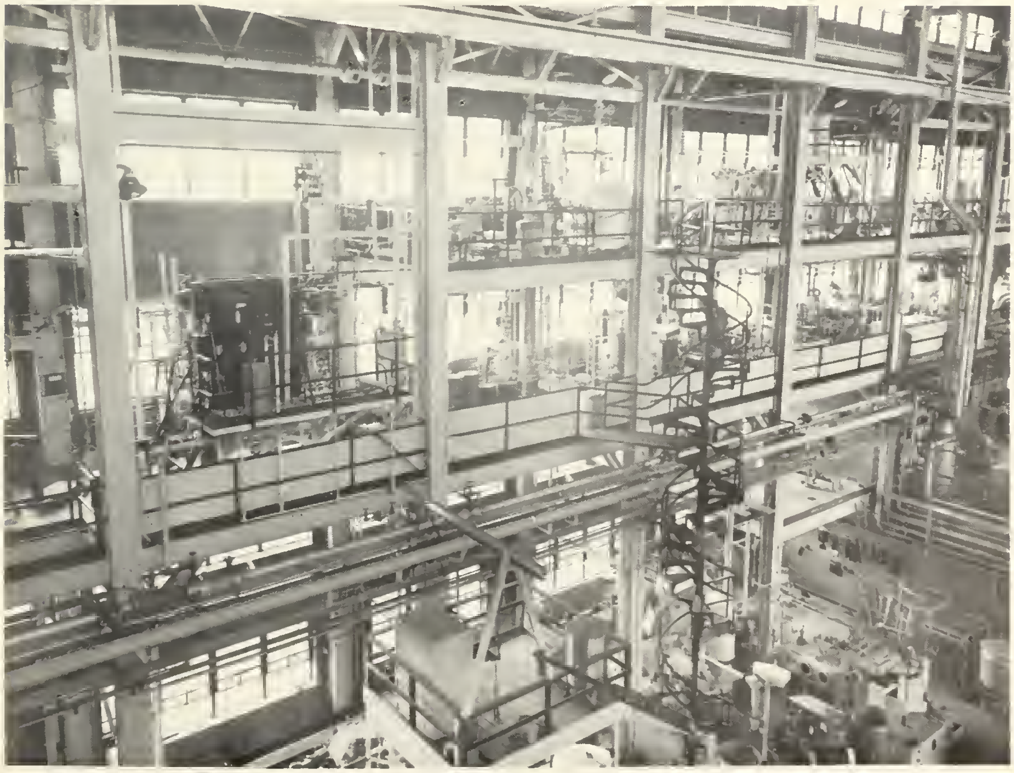


Figure 3.--Mezzanines of main pilot plant.

Figure 4.--Alcohol pilot plant.

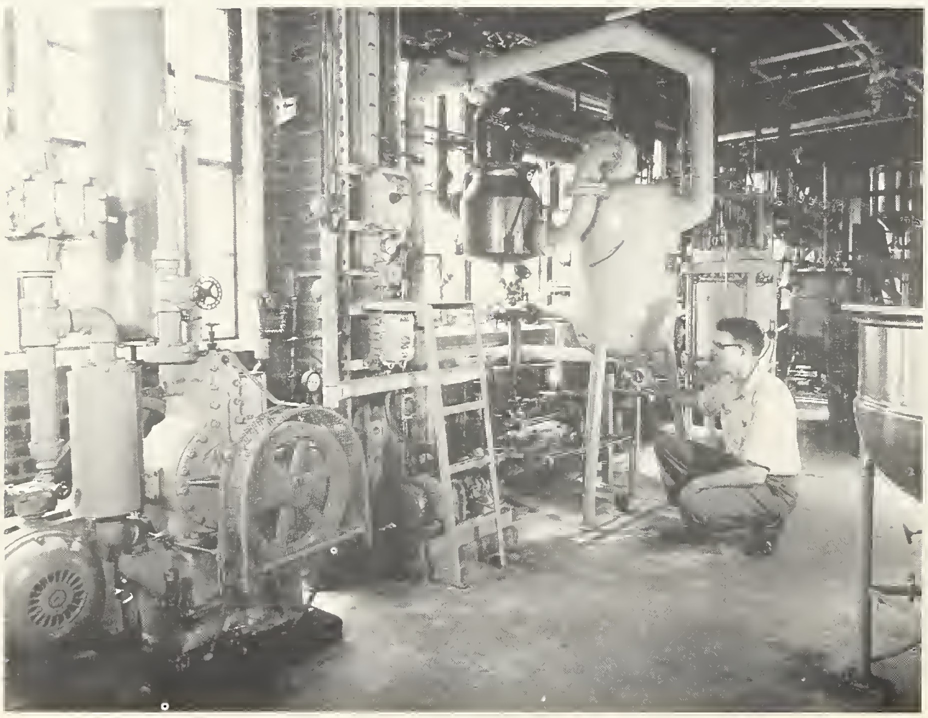


Figure 5.--Vegetable oil deodorizer in oil-extraction plant.

Figure 6 is a vertical vat fermentor used in these experimental fermentation studies. Industrial fermentation research is another of our major fields of endeavor.

Figure 7 shows a 60-gallon stainless-steel fermentor for experimental use.

Accurate and detailed analyses in solvent and related investigations are possible with an experimental distillation unit (figure 8).

Figure 9 is a view of a small vacuum drier for general pilot use on many of the grain and chemical products that are developed.

As far as we know, our dry-milling laboratory is one of the most complete experimental laboratories now in existence. Typical milling, classifying, and analytical equipment is installed as seen in figures 10 and 11.

These units exemplify those available for pilot programs. Specific new items are added, and old ones are improved as required by current studies.

Our pilot developments must be commercialized in order to achieve the desired goals. Therefore, they must be brought to the attention of potential users without delay. This may be accomplished in one or more of the following ways:

1. Technical talks before appropriate professional societies, trade organizations, or industry advisory groups.

2. Publication in appropriate journals.

3. Supplementation of publications with special information summaries and instruction bulletins for specific and detailed evaluation.

4. Personal discussions and visits with potential and logical users.

5. Information press releases, radio and television announcements, and other general information forms.

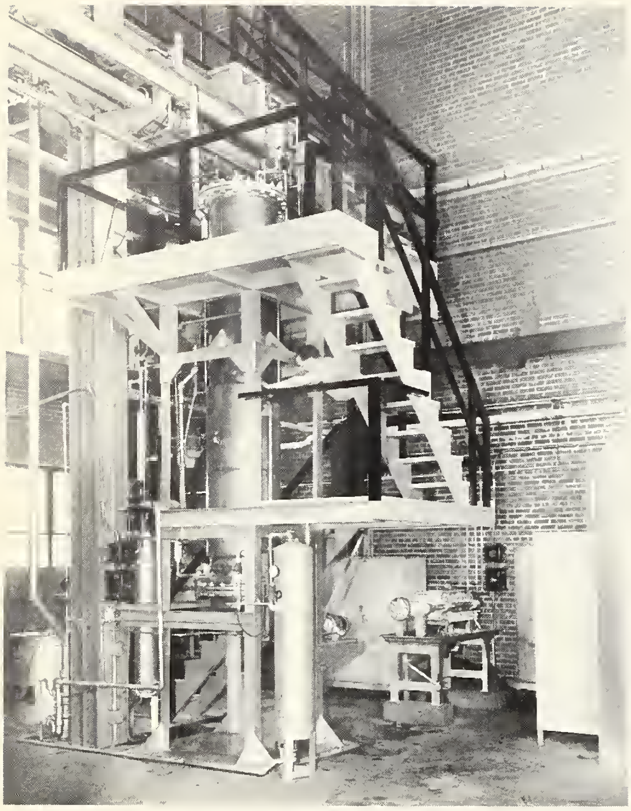
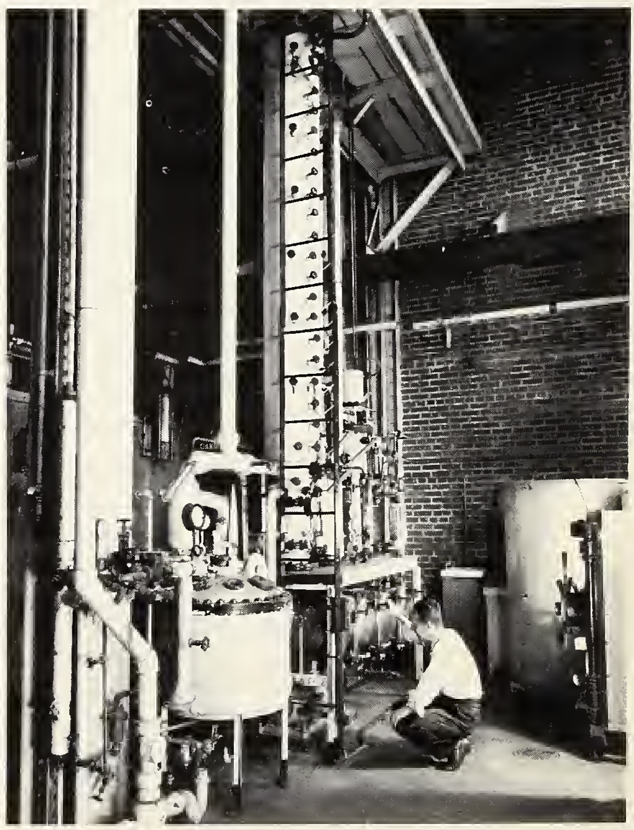


Figure 6.--Vertical vat fermentor.

Figure 7.--Stainless-steel fermentor (60 gallons).



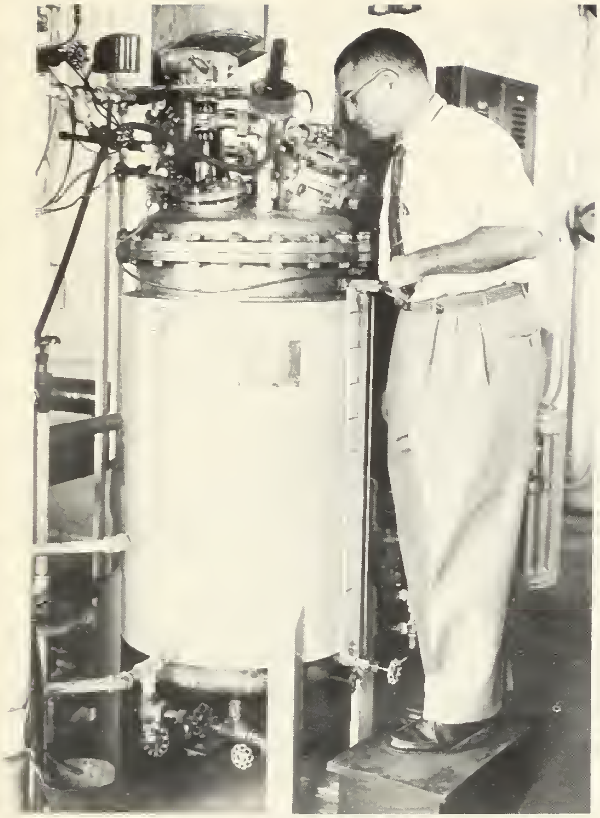


Figure 8.--Experimental distillation unit.

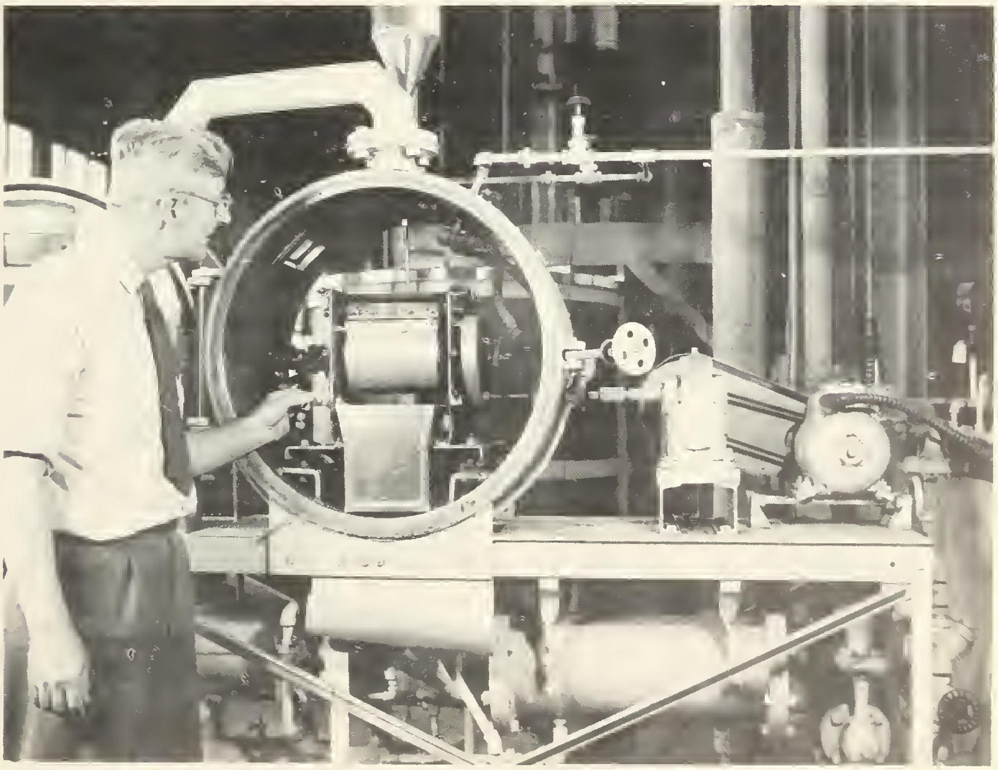


Figure 9.--Vacuum drier.

Figure 10.--Dry-milling laboratory.

Figure 11.--Dry-milling laboratory.

The old expression, "you must leave samples to get results," applies to us as much as to any industrial concern. It would be impossible for us to visualize or even estimate all the potential users of a new development. We are in the unique position of offering top-grade developments and consulting services at no charge to the user and with free licensing of all patents.

As previously mentioned, our developments are often initially presented at technical meetings and subsequently published. Reprints of these publications are available on request. If talks and published results were the only methods used to promote our developments, we would have no basis to believe that adequate coverage would be obtained or that the information would always reach the attention of the right people. Also owing to normal publishing delays, the published results usually lag behind the oral presentation by several months. Before publications are available, we usually have to depend on the initiative of those who have heard the paper to bring our developments to the attention of management and obtain the necessary work authorization. There is not always the incentive to expedite this step and company organization does not always make it easy to do so. Thus, further procedures are used to stimulate industrial interest.

Special data and instruction bulletins have been helpful in disseminating information on developments to industry. We now normally prepare such aids as soon as the product or use is announced. Copies are supplied each inquirer. The potential user can thus obtain an indication of his further interest; education time is greatly reduced; and information can be available long before actual publication of the detailed work. Correspondence Aid No. 10, "Dialdehyde Starch--Use as Additive for Wet Strength Paper" is an example (figure 12).

Over a thousand industrial representatives visit the Northern Division every year. Many companies have a regular schedule of call. This personal contact by industry itself is an excellent means of improving and demonstrating the possibilities of our developments, and at the same time, of discussing future work programs. Our visitors have the opportunity to hold discussions with our scientists and engineers who have been directly concerned with the research under discussion. Thus, a mutual understanding of each

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UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
Northern Utilization Research and Development Division
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DIALDEHYDE STARCH

USE AS ADDITIVE FOR WET STRENGTH PAPER

Dialdehyde starch, a polymeric dialdehyde developed by the U. S. Department of Agriculture, imparts wet strength to paper products either by wet end addition (*1*) or by surface application (2). Other properties such as dry tensile st... strength, and fold endurance are also improved by a... of ...

...socia...

Figure 12.--Correspondence Aid No. 10.

other's problems is realized. Although these visits are time consuming and may divert a scientist's time from research, we believe they are most important and encourage them.

A recent program adopted by the Northern Division, which has already proved itself, included the studies of market development and of industrial development. By this procedure all new developments are presented to industrial management and technical staffs by trained personnel. Information sheets, samples, reprints, and supporting data are provided. Industrial contacts are maintained. Reports and followup are analyzed and coordinated. Our own scientific staff is kept informed of the importance of a development, as well as about desired needs or modifications for the ultimate producers and consumers.

Such a group is now a part of each of the four regional laboratories. They are assisted by specialists from the Department's Economic Research and Federal Extension Services who are stationed at each Division. We always have more projects to work on than we have staff or money to carry them out. Consequently, we must concentrate on those that have the greatest possibility of success and the best chance of commercialization. In these competitive times, decisions must be made early in a promising discovery on where and how to concentrate our efforts.

The following case history illustrates the sequence of events in the industrialization of one of the developments of this Division.

PRODUCT AND PROCESS DEVELOPMENT--DIALDEHYDE STARCH

Dialdehyde starch is prepared by the oxidation of starches with periodic acid. Although the basic reation has been known for many years, the cost of periodic acid was too high for commercial use. During our program to find new chemicals from starch, a new electrolytic method, in which the periodic acid was regenerated during the reaction, was developed in the laboratory and appeared to have industrial possibilities. The convenient, inexpensive electrolytic method was patented (*2*)[4], presented at a professional society meeting, and published (*1*). The basic reaction is shown in figure 13, and the initial laboratory cell in figure 14.

Within a few months following the product and process announcement in technical meeting programs and magazines, more than 100 companies requested further information and, in most instances, samples. More than 20 requested repeat and larger samples. On the basis of this interest and on indicated industrial uses in many different markets, the process was given to the Engineering and Development Laboratory for piloting. The initial pilot-plant cells (*3*) are shown in figure 15. More than 3,000 pounds of dialdehyde starch were produced in these cells.

The information resulting from these pilot studies and the continued interest expressed by industry from the favorable cost data compiled were further evidence that an expanded engineering developmental program was justified.

During the pilot period several industrial companies investigated the dialdehyde starch process, and their representatives visited us. Several companies licensed the process for possible production, and several indicated interest as possible consumers. There was little doubt that a new processing technique for starch and new chemicals from agricultural materials had been discovered.

The expanded work initiated in our Engineering and Development Laboratory resulted in an improved and simplified production method that yielded a more uniform product, provided easier control, and was operated at a lower cost. We also contracted with Newark College of Engineering to design and build a commercial-scale electrolytic cell.

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4 Italic numbers in parentheses refer to Literature Cited, p. 24.

Preparation of Dialdehyde Starch

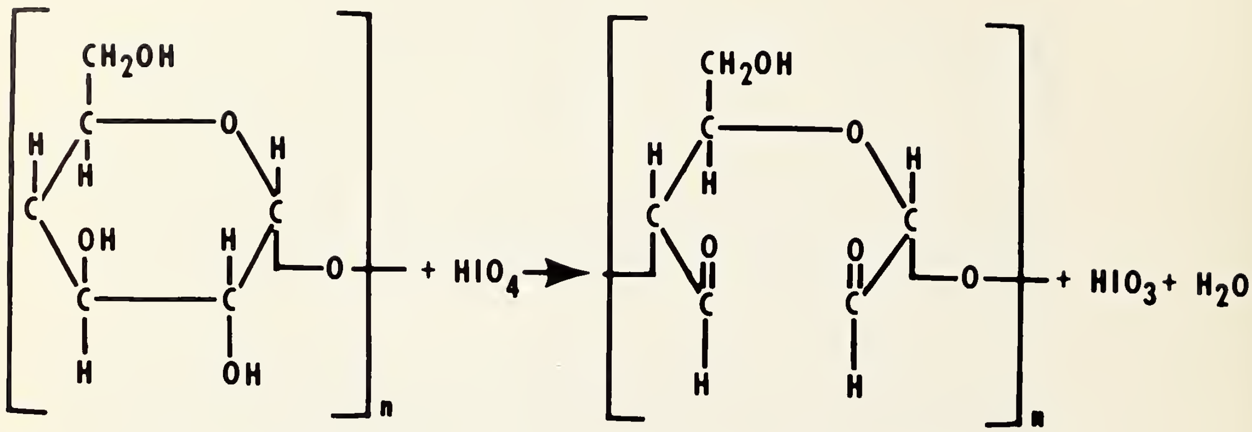

Figure 13.--Basic reaction of dialdehyde starch.

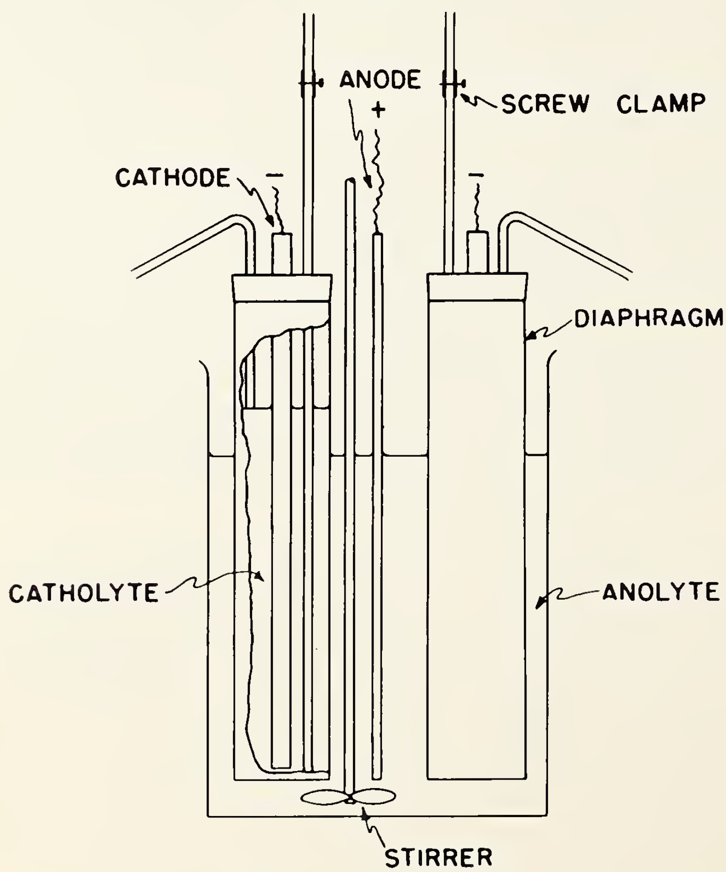

Figure 14.--Initial laboratory cell of dialdehyde starch.

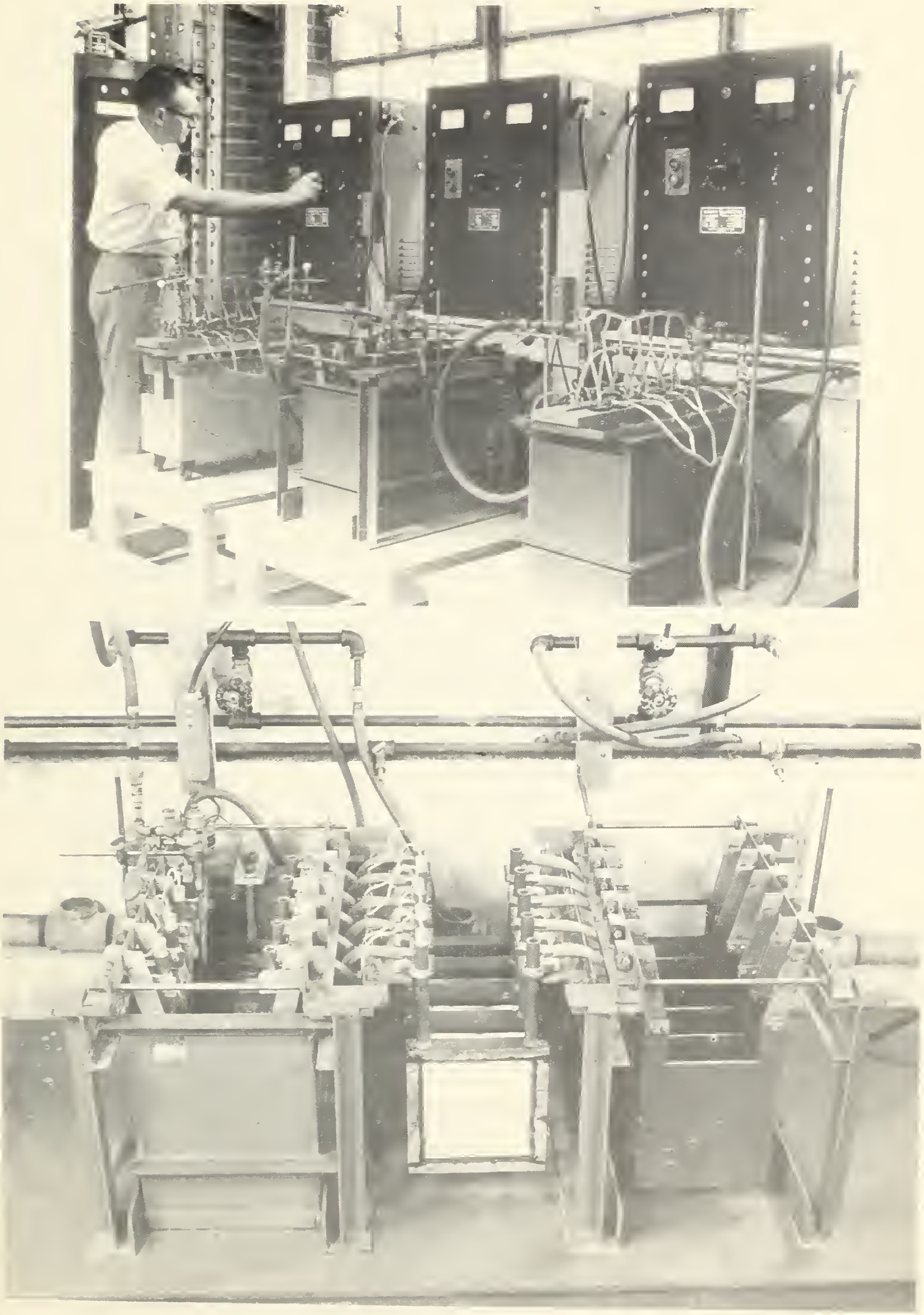

Figure 15.--Initial pilot-plant cells of dialdehyde starch. Top, general view. Bottom, detailed view.

Concurrently with results from the continuing investigations, a commercial plant (4) was designed, a preliminary flowsheet drawn(figure 16), and the plant costed.

Capital investment estimates and product cost estimates were then prepared (tables 1 and 2), and possible future markets determined. Potential uses were as follows:

- Wet-end paper additives
- Leather-tanning agent
- Reconstituted tobacco additive
- Adhesives
- Gelatin-hardening agent
- Textile-sizing agent
- New chemical raw material

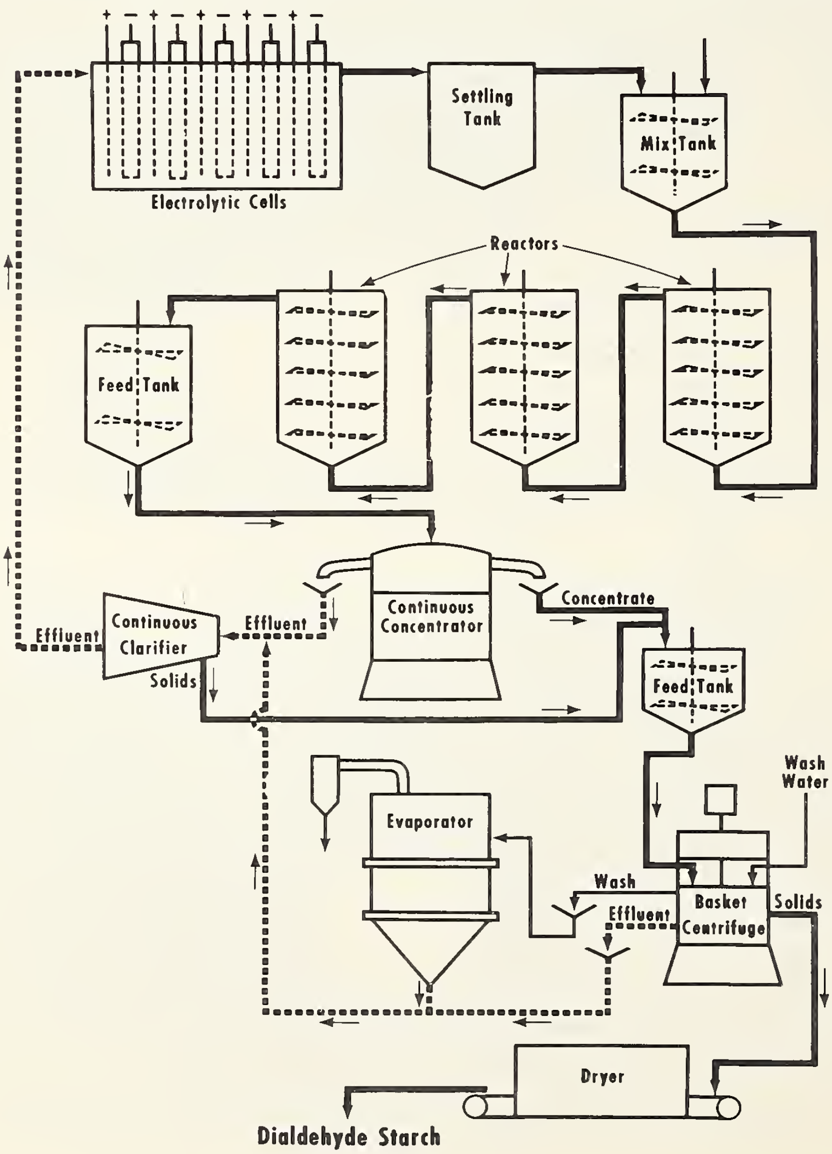

Figure 16.--Flowsheet for proposed commercial dialdehyde starch continuous plant.

TABLE 1.--Estimated fixed capital investment of dialdehyde starch plant

[Annual capacity 10 million pounds product at 11 percent moisture, 95 percent oxidation]

Item	Estimated delivered cost
	Dollars
Rectifiers, 1,600-kv.-amp., at $110/kv.-amp.	176,000
100 Electrolytic cells	300,000
Bus bars, accessories, distribution, switch gear, etc.	150,000
Heat exchangers, 150 sq. ft., two at $3,000	6,000
Mixing tanks for starch oxidation, 4,000 -gal., jacketed, agitated, 5 hp. each, vinyl-clad, four at $7,500	30,000
Continuous centrifuge, nozzle-type, 15 hp.	15,000
Basket centrifuges, 48-in. diameter baskets X 24 in. high, rubber lines, 15 hp., four at $15,000	60,000
Rotary dryer, type 304, stainless steel	50,000
Hammer mill, 20 hp.	5,000
Bagger	2,000
Evaporator, triple effect, 600 sq. ft.	90,000
Deionizing installation, 2,000 gal./hr.	25,000
Strainers for centrifuges and iodine oxidation unit, four at $2,000	8,000
Paraperiodate precipitation tank, 15,000 gal., agitated and jacketed, 20 hp., vinyl-clad	22,000

Table 1.--Continued.

Pumps, 25 required	25,000
Instruments	25,000
Conveyors	5,000
Storage bins	5,000
Feed tank for cellhouse feed, 12,000-gal., agitated, 10 hp., vinyl-clad	12,000
Feed tank for starch oxidation, 4,000-gal., vinyl-clad	4,000
Starch slurry feed tank, 4,000-gal., agitated, 5 hp., vinyl-clad	6,000
Feed tank for basket centrifuges, 1,000-gal., agitated, 2 hp., vinyl-clad	2,500
Feed tank for evaporator, 4,000 gal., agitated, 5 hp., vinyl-clad	6,000
Iodine solution tank, jacketed, agitated, 2 hp., vinyl-clad, 500-gal.	2,000
Storage tank for finished periodic acid, 1,000-gal., agitated, 2 hp., vinyl-clad	2,500
Equipment, delivered	1,040,000
Installation of equipment	260,000
Piping, wiring	300,000
Other construction costs	300,000
Contingencies, engineering, and contracting fees	500,000
Building, 265,000 cu. ft.	200,000
Land and improvements	50,000
Fixed capital investment	2,650,000

TABLE 2.--Estimated production cost for manufacturing dialdehyde starch

[Annual production 10 million pounds of product at 11-percent moisture, 95-percent oxidation; 24 hours per day, 350 days per year; 25-hour operation = 28,600 pounds of product]

Item	Daily cost	Cents per pound of product
	Dollars	
Raw Materials		
Starch slurry, 26,000 lb. dry starch at 5.0¢	1,305.00	
Crude iodine, 283 lb. at 95¢	268.85	
NaOH, 800 lb. at 6¢	48.00	
Na_2SO_4, 65 lb. at 10¢	6.50	
H_2SO_4, 700 lb. at 2.5¢	17.50	
Total cost of raw materials	1,645.85	5.75
Utilities		
Steam 100,000 lb. at 75¢ 1,000 lb.	75.00	
Water 900,000 gal. at 7.5¢ 1,000 gal.	67.50	
Electricity 38,400 kw.-hr. at 0.7¢ kw.-hr.	268.80	
Total cost for utilities	411.30	1.44
Miscellaneous Factor Supplies and Expenses	75.00	0.26
Labor and Supervision		
Operators 6 shift at $2.50/hr.	360.00	

Table 2.--Continued.

Helpers 3 shift at $2.00/hr.--	144.00	
Foreman 1 shift at $2.75/hr.--	66.00	
Laboratory technician,$20.00/day	60.00	
Superintendent, $38.40/day------	38.40	
Overhead (Social Security, pensions, vacations, etc.)----	99.30	
Total labor cost-------------	797.70	2.68
Maintenance		
Building and land, 2%/yr. of $250,000---------------------	14.30	
Equipment, 5%/yr. of $2,400,000-	342.90	
Total maintenance cost--------	357.20	1.25
Fixed Charges		
Depreciation		
Building 5%/yr. of $250,000-----	35.70	
Equipment 10%/yr. of $2,400,000-	685.70	
Taxes and insurance, 3%/yr. of $2,650,000-	227.10	
Total fixed charges-----------	948.50	3.32
Working capital charge, 5%/yr. on $192,000-----------------------	27.40	0.10
Replacement of diaphragms three times/yr. at $22,500------------	193.00	.67
Replacement of anodes one time/yr. at $14,000---------------------	40.00	.14
Total production cost-----------		15.61

In addition to our own studies, outside contracts were made with the Institute of Paper Chemistry and Armour Leather Company to make application studies.

Simultaneously with our work and equally as important, two major companies (Miles Chemical Company and Abbott Laboratories) announced they were producers of dialdehyde starch and could increase their production as the market justified. They have both embarked on development and commercialization programs. These have included magazine announcements, advertisements, brochures, and information sheets. They have also developed new uses and process revisions, which have been patented by the individual companies.

With industrial adoption, our own work is reduced. Sample requests are now referred to the manufacturers. We expect to write "successful completion" to the basic project soon.

A side development of the dialdehyde starch story has been the interest by industrial companies in the electrolytic oxidation process itself for other uses.

SUMMARY

The function, organization, facilities, and procedure for bridging utilization developments to industrial usage have been briefly reviewed. The example given also applies to several hundred developments that have resulted from utilization research; more than 200 have been adopted commerically. The Northern Utilization Research and Development Division alone has been granted more than 250 patents. Experience shows that our developmental efficiency is at least as high as that of comparable industrial groups and that substantially less time is required than the generally accepted 7 years from test tube to commercialization.

LITERATURE CITED

(1) Conway, H. F., and Sohns, V. E.
1959. A Simple Pilot-Plant Electrolytic Cell for Producing Dialdehyde Starch. Indus. and Engin. Chem. 51: 637-638.

(2) Dvonch, W., and Mehltretter, C. L.
1953. Electrolytic Preparation of Periodate Oxystarches. (U.S. Patent No. 2,648,629.)

(3) Mehltretter, C. L., Rankin, J. C., and Watson, P. R.
1957. Electrolytic Preparation of Periodate Oxystarch. Indus. and Engin. Chem. 49: 350-354.

(4) Pfeifer, V. F., Sohns, V. E., Conway, H. F., and others.
1960. Two-Stage Process for Dialdehyde Starch Using Electroyltic Regeneration of Periodic Acid. Indus. and Engin. Chem. 52: 201-206.

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